

Functionally Graded Multifunctional Hybrid Composites for Extreme Environments

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HTAM Program Review Arlington VA
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Synthesis, Characterization and Prognostic Modeling of Functionally Graded Hybrid Composites for Extreme Environments

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Virginia Polytechnic Institute and State University
Stanford University
University of Dayton Research Institute







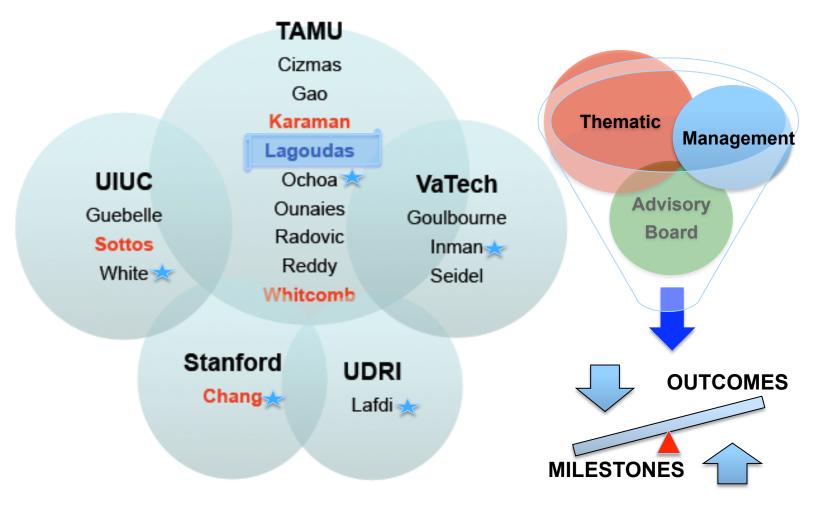








Research Team

















Goals

A comprehensive research program coupling thermalacoustic-mechanical flight loads to guide the design of multi-functional Functionally Graded Hybrid Composite (FGHC) systems with integrated sensing capabilities for extreme environments.

- multi-scale simulations
- multi-scale characterization

Target operating environment: 250 °C - 1000 °C, with a durability envelope of 1000 hours exposure at 550 °C and 300 thermal cycles















Research Thrusts

Development and Fabrication Develop multifunctional FGHC with multiple layers: a ceramic thermal barrier layer, a graded ceramic/metal composite (GCMeC) layer and a high-temperature polymer matrix composite (PMC) layer.

Multi-scale Characterization Develop and apply experimental techniques to obtain mechanical andphysical properties of GCMeC and PMC layers and of the hybrid interfaces.

Insitu NDE/SHM Integrate of SHM capabilities through networked sensor/actuator arrays, diagnostic algorithm development, control theory and fabrication optimization.

Multi-scale Modeling Develop of novel material systems for use in extreme environments: design FGHC microstructure, develop experiments and interpret data to obtain basic material properties.







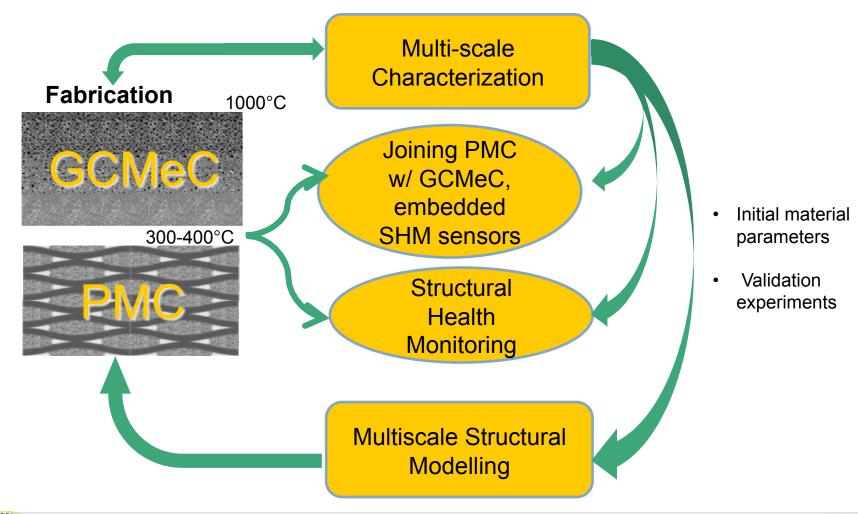








Fundamentals of FGHC

















FGHC Fabrication Team

Graded Ceramic Metal Composites (GCMeC)

Radovic (TAMU) Karaman (TAMU) Polymer Matrix Composites (PMC)

Actively Cooled PMC White (UIUC)

High
Temperature
PMC
Ounaies (TAMU)

Joining GCMeC to PMC

TAMU: Ounaies, Radovic, Karaman Lafdi (UDRI)

Embedding SHM Modules & Networks

Ounaies (TAMU), Inman (VTU), Chang (Stanford)















Fabrication and Characterization of Bulk Ceramic MAX Phase and MAX–Metal Composites







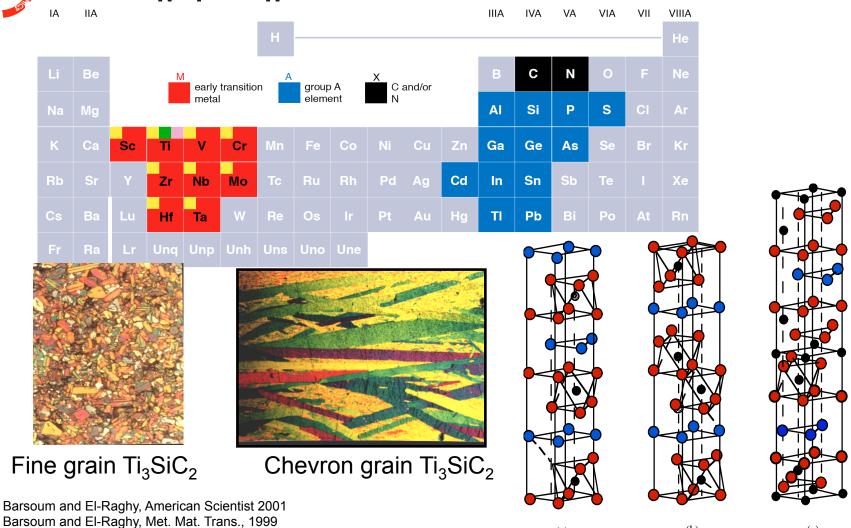








$M_{n+1}AX_n$ Phases (n = 1, 2 and 3)













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Key Technical Challenges

- Control phase fractions and distribution including gradual change in phase distribution through the thickness;
- Control over phase distribution using different processing approaches:
 - Infiltration
 - Reactive sintering
 - Co-sintering
- Interfacial integrity between metal-ceramic particles and layers
 - Both candidate alloys and ceramics have Ti.
- Long term chemical compatibility between metal and ceramic phases, and between layers with different amount of phases;
- Full infiltration of molten metal into porous MAX phase preform.









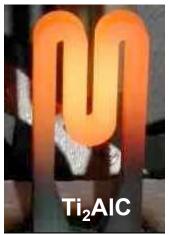


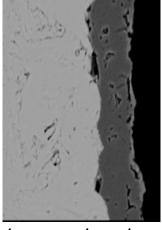




Physical Properties of MAX Phases







Cycling from room temperature to 1350°C for 8000 times forms only a few micron thick self-healing oxide layer

- Barsoum and El-Raghy, American Scientist, 2001
- 2. www.3one2.com





Ti₂AIC block after hammer blows

- · Easy to form and machine
- Stiff (320 Gpa) and tough
- Good thermal and electrical conductor
- Thermal shock and Fatigue resistant.
- Damage tolerant.
- Oxidation resistant (in air up to 1400 C).
- Low friction
- Thermally sprayable on metals for corrosion and oxidation protection.







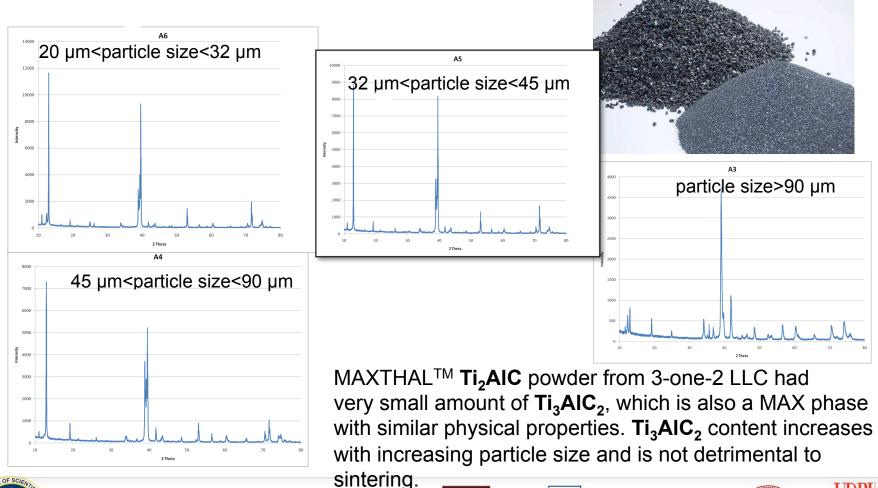








XRD Ti₂AlC Powder





AFOSR-MURI Functionally Graded Hybrid Composites









Sintering of Ti₂AIC Ceramics

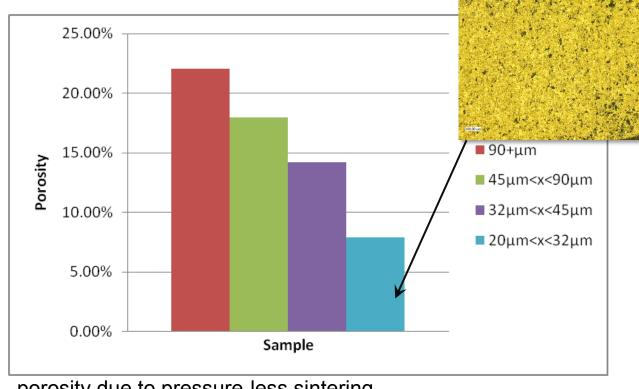
Porosity of Ti₂AIC samples after sintering at 1400°C for 4 hrs Under 96% H₂, 4%Ar

Powder Mixing

Cold Pressing

Sintering at 1400 °C - 4 hrs

Sintered Ti₂AIC



porosity due to pressure-less sintering









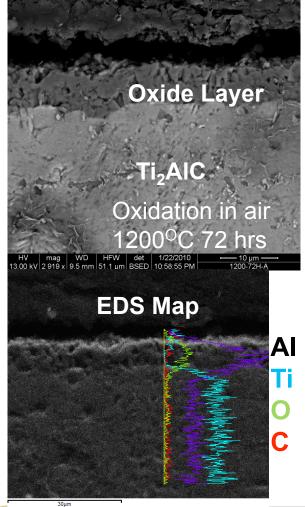


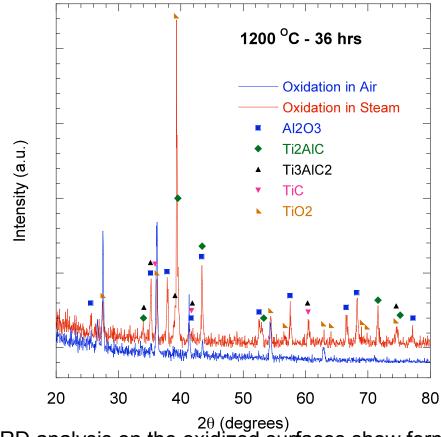




Oxidation of Ti₂AIC Ceramics

oxidation kinetics of the self-healing protective oxide on ceramic





XRD analysis on the oxidized surfaces show formation of TiO_2 and Al_2O_3 layers, which subsequently controls further oxidation







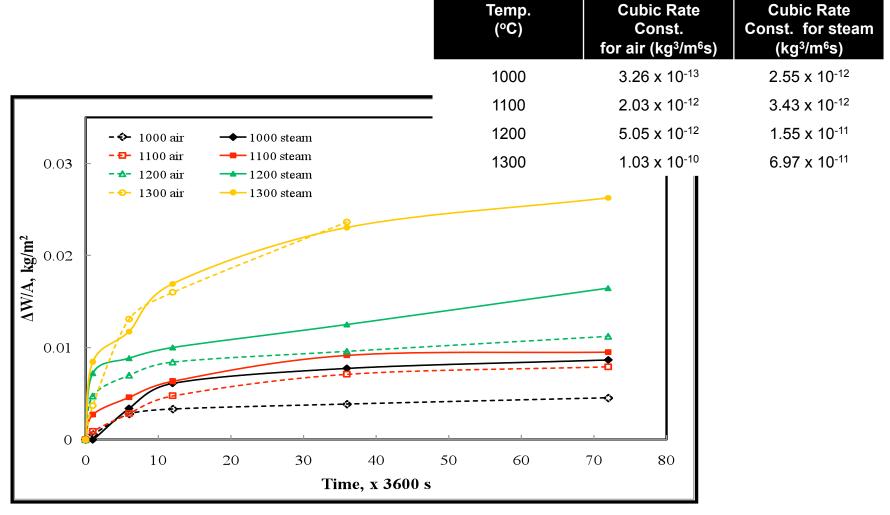








Oxidation Kinetics of Ti₂AIC Ceramics











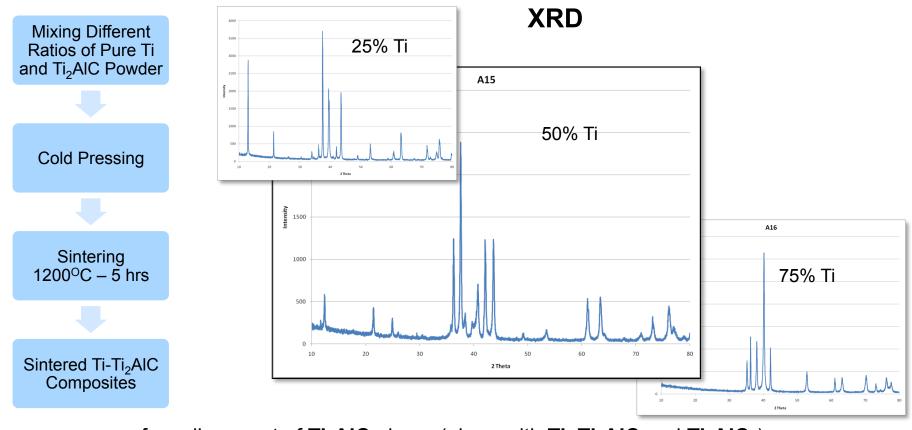






Co-Sintering of Ti-Ti₂AIC Composite

to form the ceramic-metal composite region in functionally graded structure



presence of small amount of Ti₃AIC phase (along with Ti, Ti₂AIC and Ti₃AIC₂)















Ni₅₀Ti₅₀ - Ti₃SiC₂ Composite

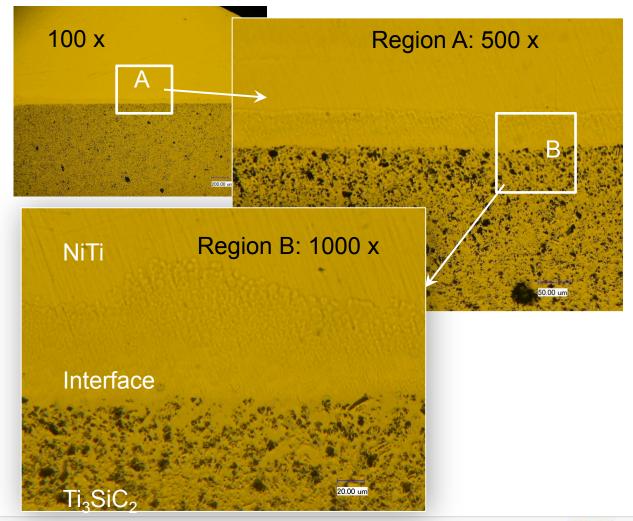
to form the ceramic-SMA composite region in functionally graded structure

Vacuum Sealing in Quartz Tube with NiTi on top of Ti₃SiC₂

Heating at 1100°C for 1 hr

NiTi Bonded together with Ti₃SiC₂, ~ 50 μm interface layer

Interface layer of about 50 micron thick formed









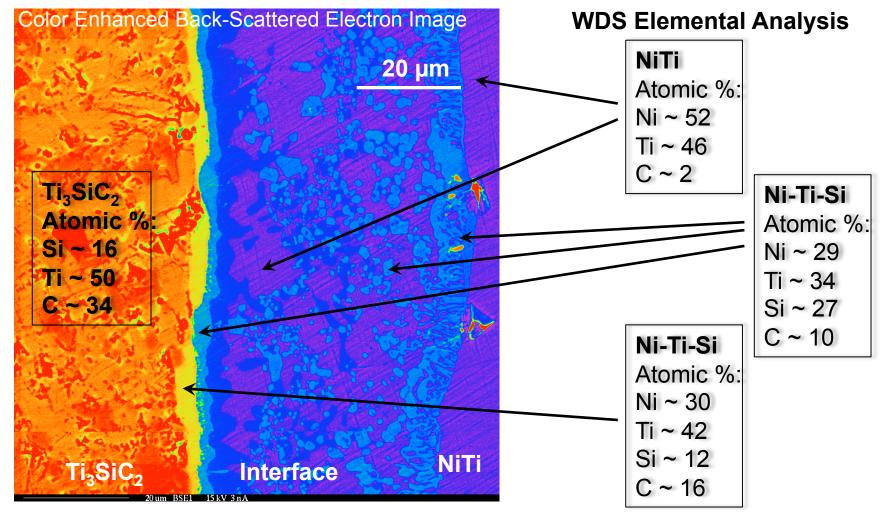








Interface of Ni₅₀Ti₅₀ - Ti₃SiC₂









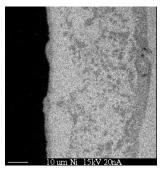




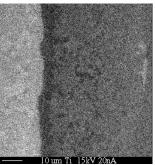




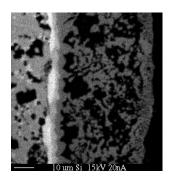
Elemental Maps: NiTi-Ti₃SiC₂ Interface



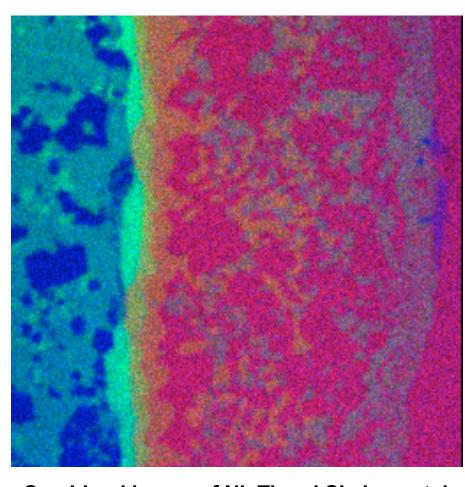
Ni



Ti



Si



Combined image of Ni, Ti and Si elemental maps (NiTi reacts with Si, from Ti₃SiC₂, to form the interfacial layer)















Research in Progress

- Sintering at different temperatures in a hot Press/hot isostatic press to obtain fully-dense bulk MAX phases
- Sintering of MAX phases with different amount of pore-formers at different temperatures to obtain MAX phase with controlled porosity for infiltration of metals and shape memory alloys (SMAs)
- Systematic oxidation study of bulk MAX phases in air, as well as in water vapor to understand the kinetics of protective oxide layer formation for selfhealing
- Systematic fabrication of porous metal and shape memory alloys by PM method, which will be used to form high-temperature polymer infiltrated composite for stronger polymer-metal interface















Research Focus

- (Glass, SiC, C) fiber- and fabric-reinforced PMC for high temperature
- Adhesion of Ti (grade 2) metal to high-temperature polyimide neat resin
- Modification of metal-PMC interface through vertical nanocolumns, Z-pins, and possibly surface modification through geo-polymer additions.















HT PMC Layer Processing

Polyimide-Based Composites

- Polyimides have good thermo-oxidative stability, resistance to moisture absorption, and relatively high moduli.
- Polyimides can withstand hot spikes up to two times their T_g.
- Viscosity tends to be elevated which makes processing a challenge

→ Solvent-assisted processing of the polymer to control viscosity.



Solvent-assisted Resin Transfer Molding





Solution-cast, Thermal Cure then Autoclave















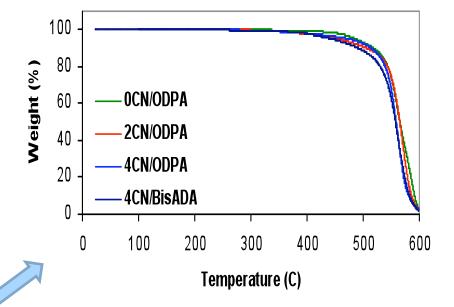
Effect of Processing on Tg

✓ Increasing nitrile content in the imide increases T_q

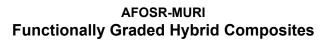
Nitrile Content	Polymer	T_{g} (°C)
0 CN		185
1 CN		217
2 CN	+\(\sigma^0\) \(\sigma^0\) \(\s	232
4 CN		260

✓ThermoGravimetricAnalysis shows that polyimides are stable up to ~450°C

Polyimide	T (C)	
	(5wt% loss in air)	
0CN/ODPA	485	
2CN/ODPA	457	
4CN/ODPA	474	











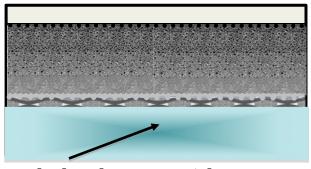




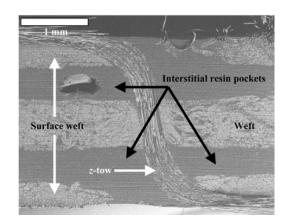




Actively-Cooled PMC (AC-PMC)



Actively cooled polymer matrix composite (AC-PMC) layer



3-D Woven Microvascular Composites. 3D orthogonally woven monolith with 56% fiber content

AC-PMC Concepts

- Short term: 2D planar array of embedded micro-channels layered within a PMC
- Long term: 3D woven PMC architectures with integrated microvascular networks with sacrificial fibers co-mingled with reinforcement tows



Multi-scale Characterization

nm Characterization of Composite Layers Graded Ceramic/Metal Matrix Composites μM Polymer/Matrix Composites Local Strain Fields/Damage Initiation Interfaces and Bonded Joints Thermal Impedance mm Interfacial Delamination Structural Performance Impact Response Vibration Analysis cm













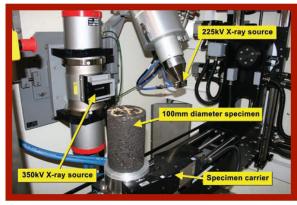


Constituent Characterization

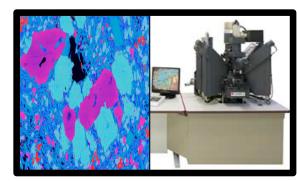
High Temperature X-Ray Diffractometer (up to 1500K)



Micro CT for non-destructive characterization of metallic and ceramic phases and porosities



Electron Microprobe Analyzer and Wave Dispersive Spectroscopy (WDS) to study compositional variations across interfaces



SEM and Orientation Imaging
Microscopy (OIM) for phase morphology,
distribution, and texture



Hermetic, beryllium dome high temp heating stage under 6x10⁻⁷ mBar vacuum







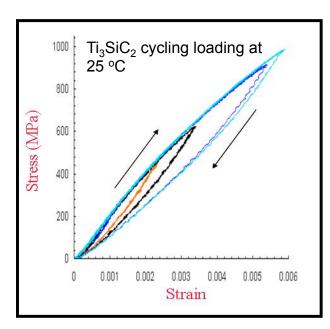




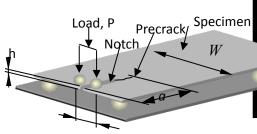


Thermo-mechanical Testing

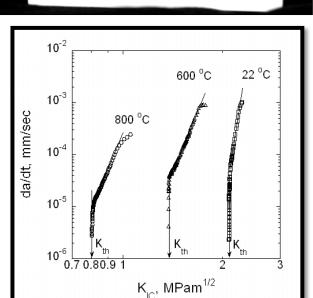
Tensile, compressive, 4-point bend, double torsion



Barsoum, Zhen, Kalidindi, Radovic and Murugaiah, Nature Materials, 2 (2003)







Fixture for high temperature double torsion.

Radovic M., Lara-Curzio E., Nelson G. Ceramic engineering and science proceedings , Vol. 27 (2007)









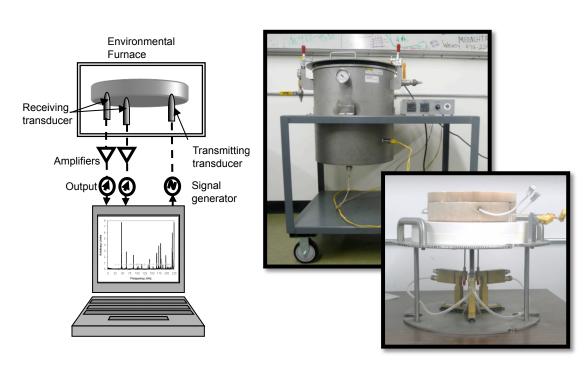


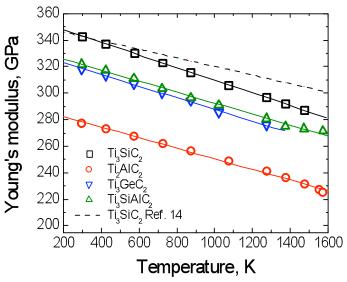




Resonant Ultrasound Spectroscopy

- Young's and Shear moduli from resonate spectrum of the material;
- One of the most accurate technique;
- Unique RUS equipment for measurements in 25-1300 °C and controlled environment developed at Texas A&M.





Radovic M. et al, JMR, Jun (2008) Radovic M. et al, Acta Mat, 54 (2006) Barsoum M.W., Radovic M. et al, Phys. Rev. Lett. 94 (2005) Radovic M., et al., Mat. Trans. A, 368 (2004)















PMC Characterization

A full suite of experimental techniques will be used to aid manufacturing development studies and mechanical/thermal performance assessment

Material	Experimental Technique	Characterization
Matrix Resin	Rheology	complex viscosity
	DSC	cure kinetics
	DMA	mechanical properties
Composite material	DSC	cure kinetics
	DMA	mechanical properties
	Optical/electron/fluorescent	material architecture
	microscopy	
	Micro-CT	material architecture
Microvascular composite	IR imaging	surface temperature field
	Fluorescent microscopy	internal fluid temp
	Micro-CT	network architecture
	Micro-PIV	flow characteristics













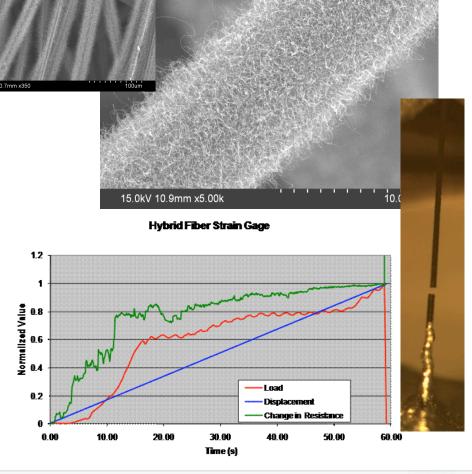


Joining GCMeC and PMC

Bonding

- vertical nano-columns
- intermediate fabric preform with vertical nano-columns
- Z-pinning technique

CNT Fuzzy Glass fiber was attached to voltmeter to measure changes in resistance as tension test progresses











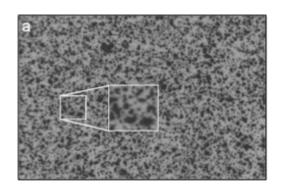


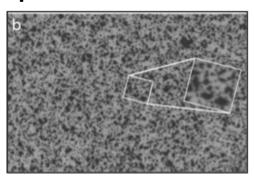




Digital Image Correlation (DIC)

Displacement-Strain Measurements

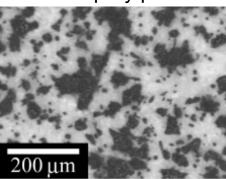




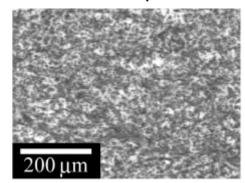
$$x'_{q'} = x_q + u_{x_p} + \frac{\partial u_{x_p}}{\partial x} \Delta x_q + \frac{\partial u_{x_p}}{\partial y} \Delta y_q$$
$$y'_{q'} = y_q + u_{y_p} + \frac{\partial u_{y_p}}{\partial x} \Delta x_q + \frac{\partial u_{y_p}}{\partial y} \Delta y_q$$

Can control resolution through speckle pattern.

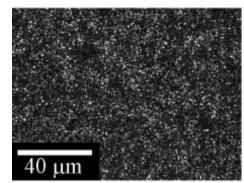
Macroscale: ±25 μm black spray paint



Microscale: ±1 μm air-brushed pattern



Nanoscale: ±10 nm fluorescent nanoparticles











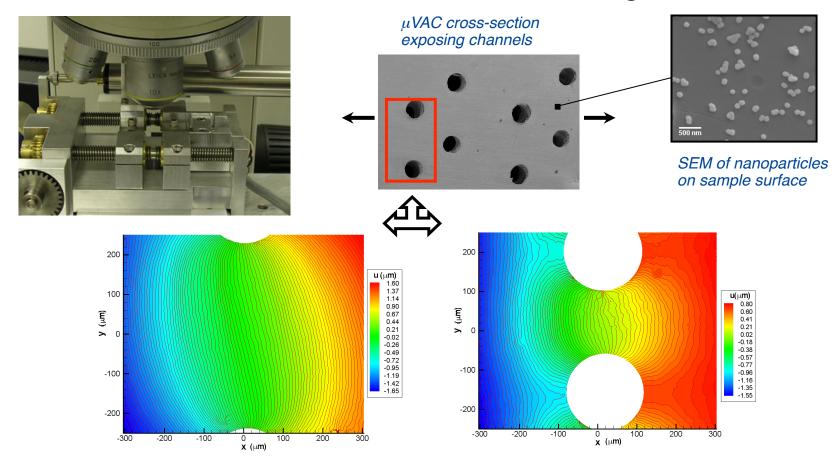






Localized Strain Measurements

Strain concentrations due to vascular cooling networks













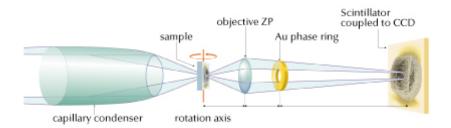




Micro and Nano X-Ray Tomography



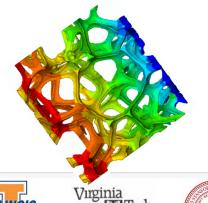
Lens based x-ray microscopy



MicroCT reveals material architecture



FEA models will be based on microscopy and micro-CT observations of functionally gradient surfaces.













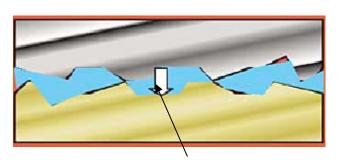


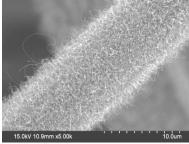


Thermal Impedance to Characterize CNT- TIM

Requirements of Thermal Interface Material

- conform to mating surface
- good wettability
- provide high conductivity path







interfacial material

Ref. Product	Thermal Impedance [°C in²/W]
Dry Test	0.160
Arctic Silver	0.013
Aremco 640	0.060
Circuit Works Grease	0.018
Omegatherm 201	0.054
PowerFilm 51	0.474

ASTM D5470 Thermal Interface Material Testing System

Sample type	Thermal Cond.W/m.K	Enhancement %
Aluminum solid	95.73	
Aluminum pieces in direct contact	8.957	
Aluminum pieces with graphite spray	37.983	324.06
Aluminum pieces with CNT/TIM	43.457	385.16









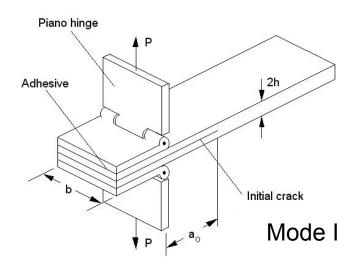


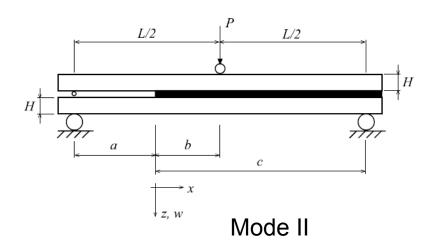




Interfacial Fracture Testing

 Assess interfacial integrity of GCMeC/PMC with DCB test for Mode-I and ENF test for Mode-II loading from room temperature to 1000 °C.





 Combined mesoscale characterization (SEM) and macro-scale characterization will provide insight in to the fracture













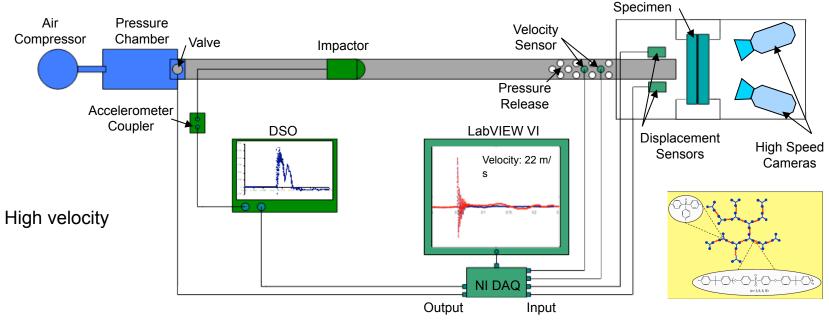


Impact & Vibration Characterization

Effect of microstructure and FGHC assembly

- Quasi-static, drop tower, and instrumented gas gun
- VT altitude chamber
- Identify damage mechanisms and dominant failure modes
- Observe interactions at the interfaces
- Strain rate dependence













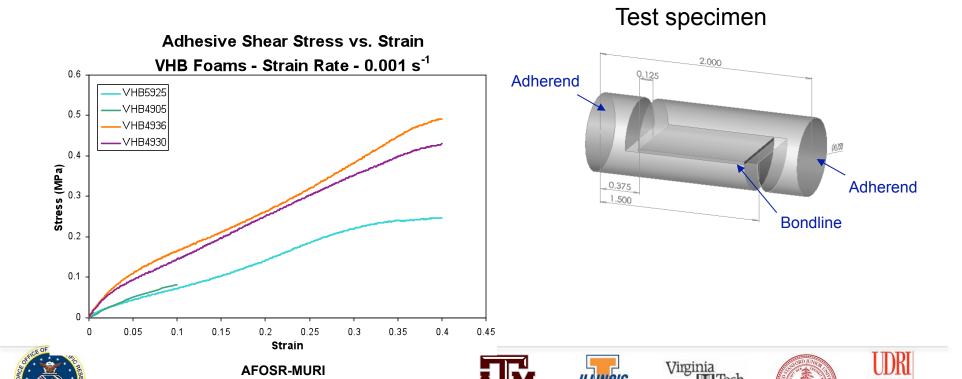






Dynamic Shear Strength Bonded Structures

- Evaluate the dynamic shear strength of interlayers and joining techniques
- Determine the static and dynamic adhesive shear strength between functional layers as a function of component properties and process conditions.



Functionally Graded Hybrid Composites



High Temperature SHM/NDE

In-situ characterization of the integrity of FGHC

Sensors & Sensing Network

Diagnostic Algorithms

Modeling

Integration & Characterization









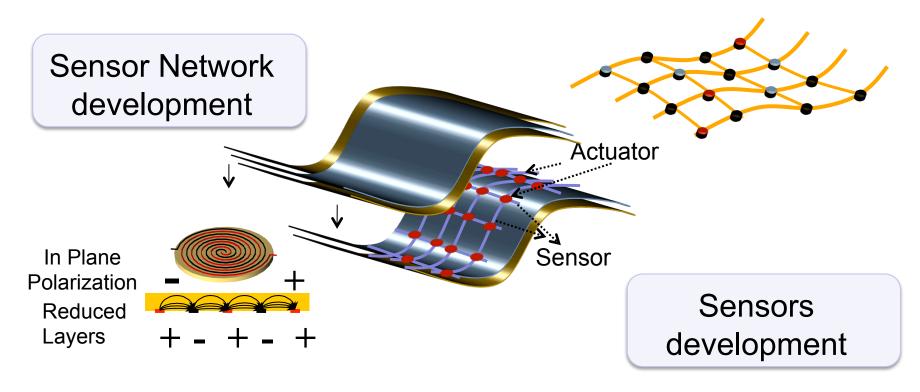






Sensors and Networks

Monitoring the health state of the hybrid composite materials during manufacturing and in service

















Sensor Development: Piezoelectrics

- High Curie Temperature piezoelectric ceramics:
 - commercially available Bismuth titanate and BST-lead titanate withT_c~500-600°C.
 - Lead titanate-based single crystals
- > Flexible 0-3 piezoelectric composites:
 - Piezoelectric inclusions in polymer matrix



βCN-PI/PZT/SWNT













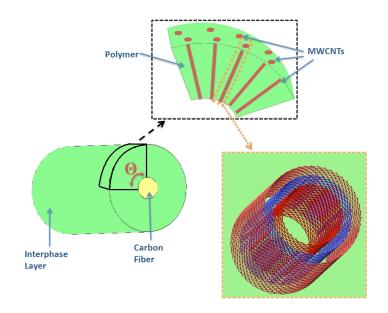


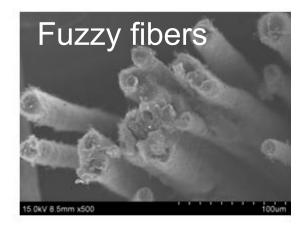
Sensors Development: Nanomaterials

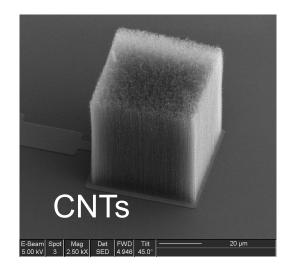
Conductivity changes



Strain, damage





















Integration and Characterization

Testing of Complete SHM/NDE System in Hybrid Composite

FGHC

Complete system development

Integration

Evaluation

Characterization

Cool side: 300-500° C

Sensor/Actuator

Hot side: 500-1000° C













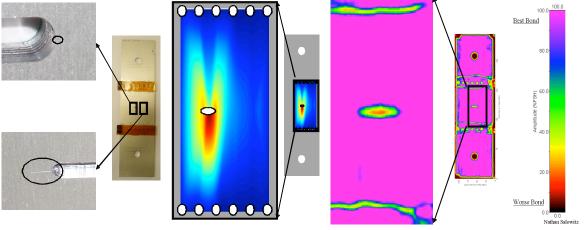


Diagnostics/Algorithm Development

- •Adapt algorithms to account for extreme temperature changes
- Develop methods to distinguish damage types
- Focus on impact force identification methods
- Develop vibration based approach to track damage induced by impact



Hybrid material with internal (Right) and externally (left) affixed PZT SMART layer networks.



a) Hair line cracks detected by visual inspection on hybrid laminates, b) Diagnostic image by SHM detected the presence of these cracks, c) Traditional NDE.









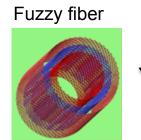


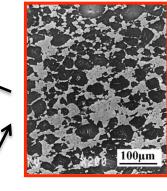




Multi-scale Modeling of FGHC

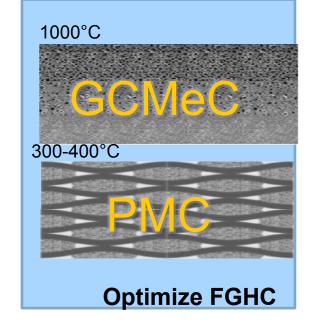
Wide Range of Scales

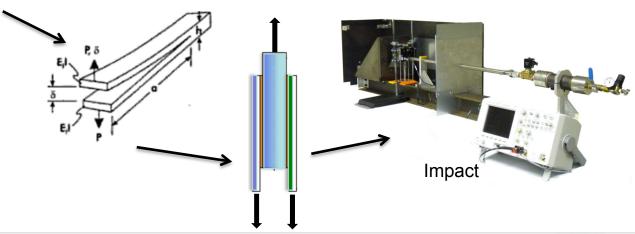


























Modeling Goals

- Predict performance of material and components
- Develop strategies for joining parts
- Expedite mechanical and thermal design
- Define in-flight mechanical and thermal loads









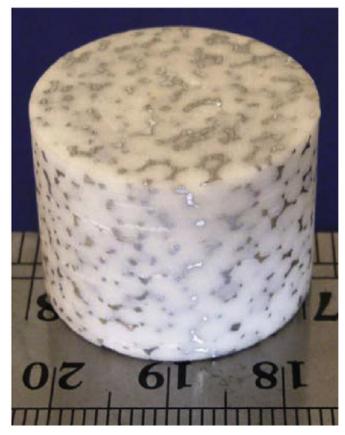




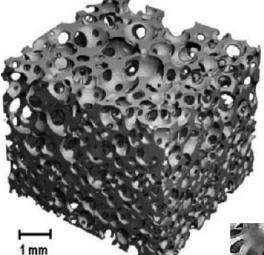


GCMeC Interpenetrating Phase Composite

Preform is random open-cell foam



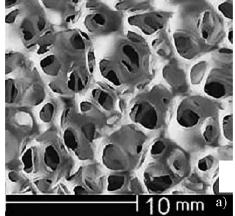
(Jhaver and Tippur, MSE-A, 2009)



SEM micrograph of Al₂O₃ preform



(Colombo & Hellmann, *Mat. Res. Innovat.*, 2002)











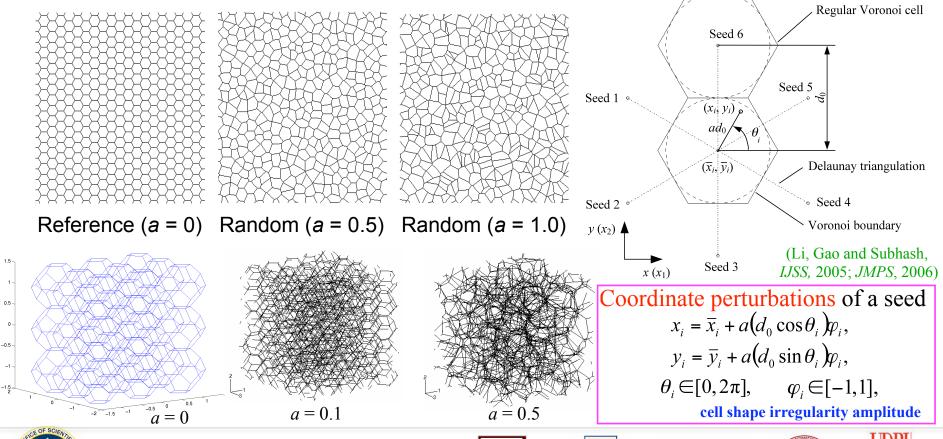




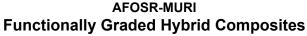


Random Cell Model

- Periodic random models Preliminary Work
- Start with reference model: structure with regular cell shapes and uniform struts
- Construct from a set of periodically located seeds using Voronoi tessellation technique













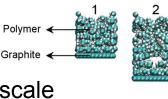


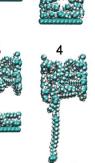


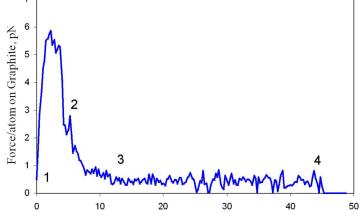


Multi-scale Progressive Damage in Complex Microstructures

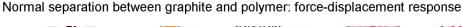
- Develop and utilize a multiscale modeling framework that will
 - allow investigation of the details of damage initiation and evolution in an FGHC consisting of a thermal barrier layer, a GCMeC and a PMC layer
 - correlate changes in nanocomposite mechanical, electrical and electromechanical properties
- Adaptive computational micromechanics tools which integrate
 - molecular dynamics
 - finite element analysis
 - homogenization
- MD to incorporate nanoscale interface effects and interphase layers into continuum level models (inelastic cohesive zone models)







Displacement of Graphite, Angstroms







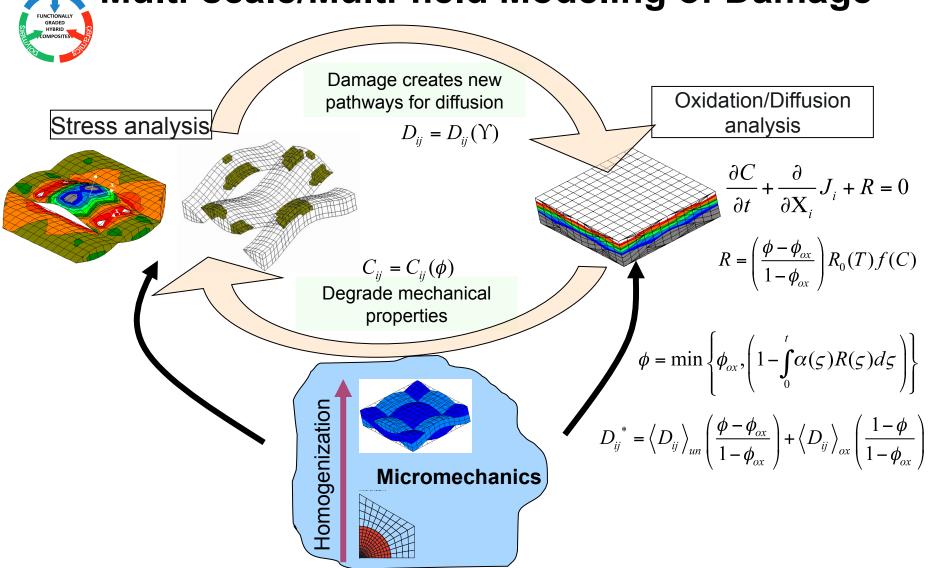








Multi-scale/Multi-field Modeling of Damage







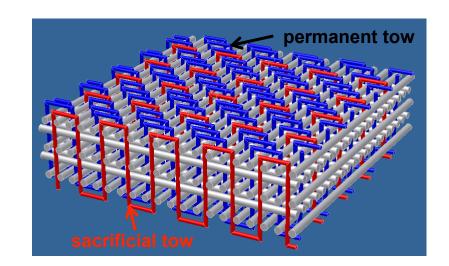






Actively Cooled Woven PMC

- Design microvascular networks embedded 2D and 3D woven PMC
- Predict homogenized thermo-mechanical response
- Technical challenges
 - Representation of composite microstructure
 - Design of network template compatible with microstructure and manufacturing constraints
 - Problem size
 - Validation with thermal and constitutive/failure assessments (White and Sottos)
 - Multiscale thermal and structural modeling













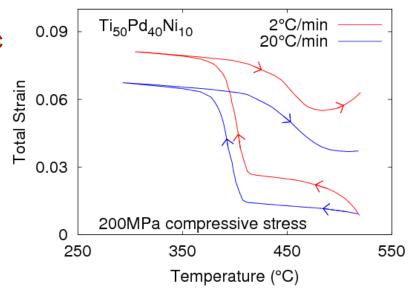




Viscoplastic Behavior of High-Temperature Active Layers

Use shape memory effect to absorb energy and induce compressive stresses in ceramic

- High temperature=> viscoplastic response becomes an important issue for the metallic constituent
- Creep is directly coupled with the transformation behavior of hightemperature SMAs



- Characterize overall creep behavior of GCMeC
- Optimize microstructure with respect to its inelastic performance
- Obtain effective creep properties by extending multiscale homogenization techniques









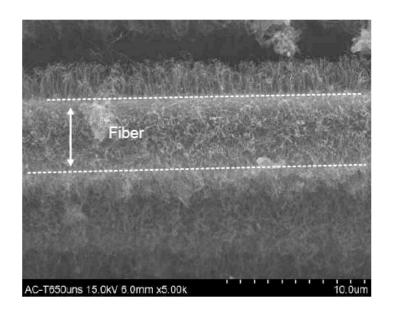






Fuzzy Fibers for Structural Health Monitoring

'Fuzzy' fibers: SiC fiber core with carbon nanotubes grown radially along fiber length



- Predict changes in electromechanical properties with damage evolution within nanocomposite ("fuzzy region")
- Optimize design of SHM sensors based on fuzzy fibers
- Integrate multiscale model for fuzzy fibers with larger length scale models for FGHC







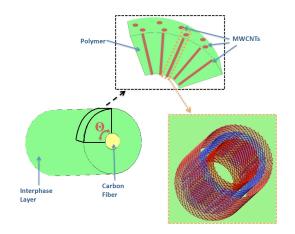


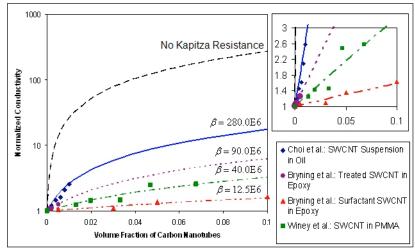






Nanocomposite-based SHM





Influence of interfacial thermal resistance on nanocomposite themral conductivity

Key Challenges Integrate

- molecular dynamics
- finite element analysis
- homogenization techniques

Accounting for

- mechanical and thermal interface effects
- nanoscale effects of electron hopping and interfacial thermal resistance
- polymer damage evolution model in nanocomposite interphase
- electromechanical properties of CNTs and its influence on fuzzy fiber SHM capabilities













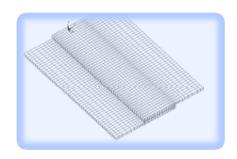


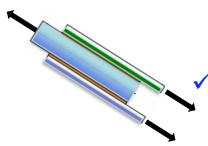
Joints for Complex Material Systems Interfaces

Systems

- MAX & Hybrid Composite
- Metal & TiGr
- PMC & Ti

- ✓ Base geometric and material heterogeneity in FEA models on microscopy and micro-CT
- ✓ Include mechanical, thermal, and oxidation effects
 - Gain insight to damage mechanisms



















Structural Response

Aero-thermo-elasticity

- Predict aero-thermoelastic response
 - Canonical double-wedged wing
 - Typical hypersonic vehicle
- Evaluate thermal effects on AE response including material degradation
- Assess effect of elastic deformation on aerodynamic heating
- Evaluate impact of inertial effects in preflutter aero-thermoelastic analysis



Impact Behavior of FGHC Structures

Develop an efficient computational framework to evaluate bonded joints and FGHC plate and shell type structural components subjected to low velocity impact.















Development and Fabrication Timeline

60/6-60/9	10/09-1/10	2/10-5/10	6/10-9/10	10/10-1/11	2/11-5/11	6/11-9/11	10/11-1/12	2/12-5/12	Year 4	Year 5
					O			E		
X	X	X	X							
		X	X	X	X				X	
						0			E	
X	X	X	X	X	X					
		X	X	X	X	X	X	X	X	x
			O				E			
X	X	X	X	X	X	X		X	X	
			X	X	X	X	X	X	X	X
								X	X	X
						0			E	
	X	X	X	X	X	X		X	X	X
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Note that **O** implies delivery of fabricated specimens and models for 1st generation SHM integrated PMC-AC, PMC-HT, GCMeC, and FGHC where as **E** implies second generation enhancementslessons learned.















Multi-scale Modeling Timeline

	60/6-60/9	10/09-1/10	2/10-5/10	6/10-9/10	10/10-1/11	2/11-5/11	6/11-9/11	10/11-1/12	2/12-5/12	Year 4	Year 5
Multiscale Modeling FGHCs and Joints											
(Whitcomb, Reddy, Cizmas, Gao, Lagoudas, Seidel, Geubelle, Inman, Ochoa)											
Design of Material Architectures					O				E		
Micromechanics based modeling of GCMeC	X	X	X	X	X						
Multiscale optimization of AC-PMC and HT-PMC	X	X	X	X	X	X	X	X	X	X	X
Modeling of progressive damage in complex microstructures		X	X	X	X	X	X	X	X	X	X
Multiscale modeling of nanocomposites-based SHM	X	X	X	X	X	X	X	X	X	X	X
Impact behavior of plate and shell FGHC structures				X	X	X	X	X	X	X	X
Aeroelastic modeling of FGHC structural response	X	X	X	X				X	X	X	

Note that **O** implies delivery of fabricated specimens and models for 1st generation SHM integrated PMC-AC, PMC-HT, GCMeC, and FGHC where as **E** implies second generation enhancements-lessons learned.















Insitu NDE/SHM Timeline

	60/6-60/9	10/09-1/10	2/10-5/10	6/10-9/10	10/10-1/11	2/11-5/11	6/11-9/11	10/11-1/12	2/12-5/12	Year 4	Year 5
In situ NDE/SHM for FGHCs (Chang, Inman,Lafdi, Ounaies,Goulbourne, Seidel)					o					E	
Fabricate polymer film sensor arrays with micro, nano ceramic PZT fillers	X	X	X	X	X	X	X	X			
Fabricate polymer films with aligned & dispersed CNTs for strain sensing		X	X	X	X	X	X	X	X		
Fabricate silicon carbide network to accommodate sensors arrays			X	X	X	X	X	X	X	X	
Develop diagnostics/algorithm		X	X	X	X	X	X	X	X	X	X

Note that **O** implies delivery of fabricated specimens and models for 1st generation SHM integrated PMC-AC, PMC-HT, GCMeC, and FGHC where as **E** implies second generation enhancements-lessons learned.















Multi-scale Characterization Timeline

	60/6-60/9	10/09-1/10	2/10-5/10	6/10-9/10	10/10-1/11	2/11-5/11	6/11-9/11	10/11-1/12	2/12-5/12	Year 4	Year 5
Multiscale Characterization of FGHCs											
(Sottos, Goulbourne, Ochoa, Lafdi, Ounaies, Radovic, Karaman, Lagoudas)											
Obtain Physical and Mechanical Properties of GCMeC and PMC Layers				О				E			
Microstructure and reinforcement architecture interdependence	X	X	X	X	X	X	X	X	X		
Local stress analysis and damage initiation			X	X	X	X	X	X	X	X	X
Thermomechanical and thermo-oxidative capacity		X	X	X					X	X	X
Interfaces and Bonded Joints						0				E	
Evaluate GCMeC/PMC joint shear strength, delamination resistance				X	X	X	X	X	X	X	X
Characterize thermal impedance of FGHC joints						X	X	X	X	X	
Assess integrity of micro-vascular network, sensor arrays			X	X	X	X	X	X	X	X	X
Structural Performance							0				E
Impact response of PMC, GCMeC, FGHCs with SHM network					X	X	X	X	X	X	
Determine modulus and damping parameters of FGHCs							X	X	X	X	

Note that **O** implies delivery of fabricated specimens and models for 1st generation SHM integrated PMC-AC, PMC-HT, GCMeC, and FGHC where as **E** implies second generation enhancements-lessons learned.















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